

**Los Alamos National Laboratory
Environmental Restoration Program
Standard Operating Procedure**

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**Calibration and Alignment of
The Siemens Diffractometers**

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CALIBRATION AND ALIGNMENT OF THE SIEMENS DIFFRACTOMETERS

1.0 PURPOSE

The purpose of this procedure is to describe the methods, procedures, and documentation required to align and calibrate the Siemens D500 X-ray powder diffractometers.

2.0 SCOPE

2.1 Applicability

This procedure applies to Siemens D500 X-ray diffractometers used to obtain analyses for the Environmental Restoration (ER) program. Due to safety considerations, the machine custodian is solely responsible for performing machine alignment. The ER user and the machine custodian performing calibration have the responsibility to adhere to this procedure.

2.2 Training

Only the machine custodian shall perform alignment of the instruments. Other personnel who have the technical training necessary may be certified to use this procedure. Reading and understanding this procedure as well as the six procedures in Section 1.0, General Instructions shall constitute training to this procedure.

3.0 DEFINITIONS

- A. Machine Custodian: The person who is in charge of an analytical X-ray instrument and who is responsible for the maintenance and safety of the instrument.
- B. Shorting Plug or Key: The plug (θ - 2θ machine) or key (θ - θ machine) used to bypass the safety circuits to allow alignment. To be used only by the machine custodian.
- C. TTK heating stage: The Anton-Paar heating stage that can be mounted on either the θ - θ or the θ - 2 diffractometer.
- D. NIST: National Institute of Science and Technology.

4.0 BACKGROUND AND/OR CAUTIONS

Calibration and, if necessary, alignment of the Siemens X-ray diffraction equipment must be performed on a periodic basis in order to assure that all data collected on the instruments yield accurate d-spacings. The calibration described here involves only d-spacings. Calibration may use any well-characterized d-spacing standard that is traceable to NIST, such as their standard reference materials. It is not necessary that the diffractometer have no error in 2θ . Calibration is performed to determine the magnitude and direction of any error in 2θ so that appropriate

calibration curves may be used when obtaining d-spacings. Alignment is performed only when calibration data reveal a significant error in alignment, usually after modification of the instrument (e.g., installation of new X-ray tube, installation of alternate sample holders, installation of the TTK).

Certain aspects of the alignment procedure require that one bypass the interlocks to work on the instrument with the X-ray beam on. The use of extreme caution and safety equipment such as lead-lined gloves is mandatory.

Highly accurate d-spacing measurements are not crucial to qualitative phase identification or quantitative phase analysis. However, it is useful and good laboratory practice to have a well-aligned diffractometer. In general, errors less than $0.02^\circ 2\theta$ are desirable, but errors up to $0.10^\circ 2\theta$ are acceptable in many cases. The Siemens DIFFRAC5000 software package will accept calibration data of virtually any magnitude so that relatively large errors will be corrected. It is recommended that any calibration yielding errors of greater than $0.02^\circ 2\theta$ be followed by alignment to reduce the errors below $0.02^\circ 2\theta$. If calibration is not performed, calibration data should be documented with an explanation why further alignment was not necessary or practical.

There are few sources for error that would not be readily detected. A potential situation where minor error could occur is when the linear absorption coefficient or transparency of a sample being run is not similar to that of the standard used to calibrate the instrument. In this case, if accurate d-spacings were desired, the instrument should be calibrated with a standard of appropriate linear absorption coefficient or an analytical correction should be performed to correct for transparency effects.

The entry in the Daily Activity Log (see SOP-01.04) and the calibration sticker placed on the instrument shall constitute evidence that the procedure has been implemented and satisfactorily accomplished.

5.0 EQUIPMENT

- NIST SRM 640a or 640b Si Powder d-spacing Standard.
- Granite surface plate or precision flat plate.
- Height Gauge.
- Dial Indicator Gauge.
- Precision Right-Angle Block.
- Assorted Slits.
- Strip Chart Recorder.
- Assorted Screw Drivers and Wrenches.
- Shorting Plug or Key.
- Siemens DIFFRAC5000 software package.
- LCLSQ, lattice parameter refinement program.

6.0 PROCEDURE

Note: Only NIST traceable standards such as NIST SRM 640b shall be used to calibrate or check the calibration of the Siemens X-ray powder diffractometers.

Deviations from this procedure shall be fully documented on Daily Logs explaining the deviation and the effects it may have on the resulting work.

6.1 Calibration of Diffractometers

As required, either by time elapsed since last calibration check or by a change in the instrument or sample holder geometry, the instrument shall be calibrated against a certified standard reference material (SRM), such as NIST SRM 640a or 640b (Si powder), or against a secondary reference standard traceable to an NIST standard.

6.1.1 The calibration standard may be mounted either in a cavity or as a thin film to minimize specimen transparency effects.

6.1.2 The calibration standard shall be examined on the instrument over a 2θ range practical for the instrument and standard. A recommended procedure is to examine NIST SRM 640a or 640b from $\sim 18^\circ 2\theta$ to $\sim 150^\circ 2\theta$ using 0.01 - $0.02^\circ 2\theta$ steps and counting for at least 1.0 sec per step.

6.1.3 Determine peak positions for all diffraction maxima using the Siemens FIT program in the DIFFRAC5000 software package, typically using the split-Pearson VII profile. The results of the profile refinement may be compared with the certified values for the standard to assess the magnitude of the 2θ error.

6.1.4 Input observed and theoretical peak positions into the Siemens calibration file using the DIFFRAC5000 software. Detailed instructions are in the Siemens DIFFRAC5000 manual (Ref A).

6.2 Alignment of the Diffractometer

If a calibration check reveals significant errors in alignment (2θ errors generally greater than $0.02^\circ 2\theta$), an alignment of the diffractometer should be performed. If only some aspects of the instrument have been modified, only an abbreviated alignment will be required. For example, if a new X-ray tube has been installed, only the tube height will require alignment. When the TTK stage is installed, both sample height (6.2.3) and θ zero point (6.2.5) must be checked and adjusted if necessary.

6.2.1 Specimen holder pin height. This step is performed only if the specimen stop-screws have been modified. Thus, in practice it is seldom if ever performed. 6.2.1 is not applicable to the TTK stage.

6.2.1.1 Check the three specimen stop-screws on the specimen holder for the same height. The two outer pins should be coplanar with the inner reference pin. (Item 46, Fig. 7, in reference 2.2).

Tools Needed

- 1) Granite surface plate or precision flat plate
- 2) Height gauge
- 3) Dial indicator gauge
- 4) Precision right-angle block.

6.2.1.2 Set the level of the specimen holder using the bar by the inner stop-screw.

6.2.1.3 Set the dial indicator height using the bar or inner screw.

6.2.1.4 Before adjusting stop-screws, the locking screws should be loosened. There is one for each stop-screw.

6.2.1.5 When the height of the three stop-screws is set, retighten the locking screws.

6.2.2 Install the specimen holder on the specimen carrier using the push rod which is built into the carrier. Use the flat surface of the rod and try to make the holder parallel to the rod. This step is performed only if the specimen holder has been removed from the specimen carrier. This step is not applicable to the TTK stage.

6.2.3 Check the specimen surface (this can be done without shorting plug or key in). The axis of rotation of the goniometer is on the specimen surface when the maximum intensities appear at $\theta = 0^\circ$ (front side) and 180° (back side). Note that the maximum intensities are approximately the same. This step is done only with the θ - 2θ diffractometer and only if the specimen height has been modified. In practice, this is seldom if ever performed.

6.2.3.1

Slit positions:	I	II	III	IV
Slit:	1°	1°	---	.018°

(These are final slits - can start with wider slits, particularly #IV.)

6.2.3.2 Install glass alignment slit.

6.2.3.3 Insert an absorber plate into the direct beam to limit counts to below ~40,000 counts/sec. Set tube power to minimum (~7kV and ~3mA).

6.2.3.4 Find the maximum intensities by adjusting θ and 2θ independently and alternately on front ($\theta = 0^\circ$). Record 2θ and full scale counts.

6.2.3.5 Find the maximum intensities as above on back side (θ at 180°). This maximum intensity should be about the same value as the front side ($\theta = 0^\circ$). If intensity is different, move holder up and down - if you can't get it, the three pins are not in the same plane and 6.2.1 should be performed.

6.2.3.6 It is not important at this time that the θ and 2θ counting mechanisms read zero, but note only the 2θ values.

6.2.3.7 Ignore the θ value all together. The 2θ values only need to be the same, for θ front and back.

6.2.3.8 Compare the 2θ numbers found on front and back sides. The difference should be no greater than 0.02° . Do a θ - 2θ scan on both front and back to insure that you are aligning on the main peak.

6.2.3.9 If the difference is greater than 0.02° , the specimen holder has to be moved closer or farther away from the push rod. If the front 2θ value is larger than the back 2θ value, the holder needs to be moved towards the pin, i.e., down.

6.2.3.10 If the intensities (front and back sides) are not within $\sim 10\%$ of each other, then step 6.2.3.8 should be rechecked.

Note: The specimen surface height is set roughly on the TTK stage with the TTK alignment slit. However, the slit is too coarse to allow fine tuning of the specimen height. For the TTK and the θ - θ diffractometer (for which the sample stage cannot be rotated), fine adjustment of the specimen height is done performing multiple data collection runs and lattice parameter refinements using the program LCLSQ. LCLSQ allows refinement of a specimen displacement correction. Iterative data measurement, lattice parameter refinement, and specimen height adjustment will allow the specimen height to be adjusted precisely. Major adjustment should only be required after disassembly of the TTK stage. Adjustment of the specimen surface height on the θ - θ diffractometer should not be performed unless the specimen carrier has been disassembled.

6.2.4 Adjust tube height for maximum intensities when 2θ is reading zero. Use only the 0.018 slit in position IV and the glass slit in the sample position. Use no other slits.

6.2.4.1 Loosen the nut at top of tube slightly with small wrench in the D500 accessory kit. Loosen the allen screw at the lower back of the tube (Item 206 in Fig. 22d, reference B).

6.2.4.2 Move θ to front side.

6.2.4.3 Adjust 2θ off of maximum intensities, slightly toward zero.

6.2.4.4 Adjust tube height to get back toward maximum intensity (Item 215 of Fig. 22e in reference B).

6.2.4.5 Adjust θ so maximum intensity is restored.

6.2.4.6 Repeat above steps until you get zero 2θ with maximum intensity.

6.2.4.7 Tighten the nut and screw loosened in 6.2.4.1 above.

6.2.4.8 You should not have to set the height again unless a new tube is installed.

6.2.4.9 If the Kevex Psi detector is installed, steps 6.2.4.1 through 6.2.4.8 cannot be performed. Instead, tube-height alignment is performed using a high-angle reflection of a NIST standard such as Si. The tube height is incrementally adjusted until the high-angle reflection is located at the correct 2θ value obtained using profile refinement. Turning the allen screw on top of the tube clockwise (from above) lowers the observed peak positions.

6.2.5 With the glass slit in the sample position, adjust θ for zero with maximum intensity by means of the eccentric screw (Item 183 in Fig. 20 of reference B). As in previous 2θ adjustment, move toward zero slowly. Peak the eccentric in small steps until you get to zero. Recheck after adjusting slits (slits 0.1, 3, --, 0.018 in slit positions I, II, III, and IV, respectively). This alignment cannot be performed with the Kevex Psi detector installed, and the scintillation detector must be installed. This step needs to be performed only if the specimen carrier has been removed. This step is difficult with the TTK stage because the alignment slit is so coarse. The recommended procedure with the TTK stage is to optimize the θ zero alignment with the slit and correct for any errors using the calibration procedure.

6.2.6 Adjust slit holder for slit positions I and II. Put in the smallest slits. If there is no intensity change, the slits are properly aligned. Do the same for slit positions III and IV.

6.2.6.1 Insert the following slits into the slit holder:

Slit position:	I	II	III	IV
Slit:	0.1°	3°	---	0.018°

6.2.6.2 Remove the sheet metal shield of the slit holder and, if necessary, the Soller slits.

6.2.6.3 Loosen the locking screws (Item 232 in Fig. 25 of reference B).

6.2.6.4 Reposition the sheet metal shield (radiation protection).

6.2.6.5 Displace the slit holder by means of the two adjusting screws (Item 231 in Fig. 25 of reference B) until the ratemeter or chart recorder indicates maximum intensity. In order to turn one adjusting screw in, it is necessary to loosen the opposite screw because these are pressure screws.

6.2.6.6 Insert smallest (0.1°) slit in position II, and you should get the same intensity or you must readjust.

6.2.6.7 Loosen two bottom screws for slit III and IV holder (allen screws are right behind slit III in white painted metal). Adjust slit holder for III and IV with the smallest slits in positions III and IV.

6.2.7 Run a scan on the glass slit through zero (see Fig. 5, reference C). This step does not apply to the TTK stage.

6.2.7.1 Set up for scan:

Slit position:	I	II	III	IV
Slit:	1°	1°	1°	0.018°

6.2.7.2 Set generator to minimum power ($\sim 7\text{kV}/3\text{mA}$ for FK60-04 Cu tube), ratemeter to $\sim 2 \times 10^4$ counts/sec full scale, time constant = ~ 0.2 , absorber in direct beam, diffractometer set to step at $1/5^\circ/\text{min}$ upscale, the chart recorder set to feed at $2\text{ cm}/\text{min}$, have θ and 2θ coupled.

6.2.7.3 Start scan at $-999.500^\circ 2\theta$ (scan range is dependent on tube type).

6.2.7.4 This scan must be run automatically on the θ - θ machine, using the same conditions, starting 2θ position, and a $0.01^\circ 2\theta$ step size.

6.2.7.5 A good alignment will fulfill the following conditions:

- 1) The maximum of the primary beam must be within 0.006° of 0° .
- 2) The amplitudes of the two side maxima should not differ from one another by more than $\sim 30\%$.
- 3) The amplitude of the main maximum should be about three times as high as those of the side maximum.

6.2.8 Optimization of 2θ calibration. This is done using a certified d-spacing standard such as the NIST SRM 640 Si series and compensates for systematic errors such as sample transparency and 2θ offset.

6.2.8.1 Mount the calibration standard as in 6.1.1.

6.2.8.2 With θ and 2θ synchronized, drive to high angles (typically over $100^\circ 2\theta$) and locate a peak of appreciable intensity (one that can be easily detected). Set the X-ray tube kV and mA to typical operational values (45 kV , 35 mA).

6.2.8.3 Perform a quick automatic scan over the range of the peak and determine the observed peak position using the DIFFRAC5000 FIT program.

6.2.8.4 If the position of maximum intensity is not at the certified value, adjust the peak position by adjusting the tube height as in 6.2.4. When this step is complete, the position of maximum intensity should be at the certified value.

6.2.8.5 Refine the adjustments made in 6.2.5 and 6.2.6.

6.2.8.6 For a quick check of the quality of alignment, one or two high-angle reflections may be measured as in 6.1. After determining that alignment is sufficient, perform complete calibration as in 6.1.

6.3 Optimization and calibration of the solid-state Si detectors.

6.3.1 Perform detector energy calibration as described in Kevex 4911 user's manual (reference D).

6.3.2 Set the detector bias to 500 volts.

6.4 Data Analysis

The Siemens DIFFRAC5000 software package is used to regress all data produced or required by this procedure. The program LCLSQ is used to determine the specimen displacement error when aligning the TTK stage and can also be used to determine a specimen displacement error with the conventional stages on both diffractometers.

7.0 REFERENCES

- A. Siemens DIFFRAC5000 User's Manual, Siemens Corporation, Cherry Hill, NJ.
- B. Siemens D500/501 Diffractometer Operating Instructions, #C72000-B3476- C42-6, Siemens Corporation, Cherry Hill, NJ.
- C. Siemens Application Note #52, The New Siemens Diffractometer, Model D500.
- D. Kevex 4911 Single Channel Spectrum Analyzer User's Manual, #7257-4911.
- E. LANL-ER-SOPs in Section 1.0, General Instructions.

8.0 RECORDS

All calibration and alignment performed on Siemens diffractometers used for ER shall be documented on Daily Logs. Calibration records shall include date of calibration, standard used,

signature of the person performing calibration, the peak positions observed for the standard and the necessary corrections to 2θ . Alignment records shall include the date of alignment, the procedures performed, and the signature of the machine custodian performing the alignment. A new calibration sticker shall be placed on the front of the diffractometer following each calibration identifying when the instrument was calibrated, when the next calibration is due, and the name of the person who did the calibration.

9.0 ATTACHMENTS

N/A